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NP9 1RH

1. Your reference — SP/4165 GB

2. Patent application number *(The Patent Office will fill in this part)* — 0408234.3 — 13 APR 2004

3. Full name, address and postcode of the or of each applicant *(underline all surnames)*

UNIVERSITY OF BRISTOL
Senate House
Tyndall Avenue
BRISTOL
BS8 1TH

Patents ADP number *(if you know it)* — 7981813

If the applicant is a corporate body, give the country/state of its incorporation — UNITED KINGDOM

4. Title of the invention — PROBE FOR AN ATOMIC FORCE MICROSCOPE

5. Name of your agent *(if you have one)* — STEVENS HEWLETT & PERKINS

"Address for service" in the United Kingdom to which all correspondence should be sent *(including the postcode)*

Halton House
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LONDON
EC1N 2JD

Patents ADP number *(if you know it)* — 1545003

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Country	Priority application number *(if you know it)*	Date of filing *(day / month / year)*

7. If this application is divided or otherwise derived from an earlier UK application, give the number and the filing date of the earlier application

Number of earlier application	Date of filing *(day / month / year)*

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b) there is an inventor who is not named as an applicant, or
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Claim (s)	6
Abstract	-
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Translations of priority documents	-
Statement of inventorship and right to grant of a patent *(Patents Form 7/77)*	-
Request for preliminary examination and search *(Patents Form 9/77)*	-
Request for substantive examination *(Patents Form 10/77)*	-
Any other documents *(please specify)*	-

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Signature Stevens Hewlett & Perkins Date 13/04/04

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SARAH PERKINS 0207 404 1955

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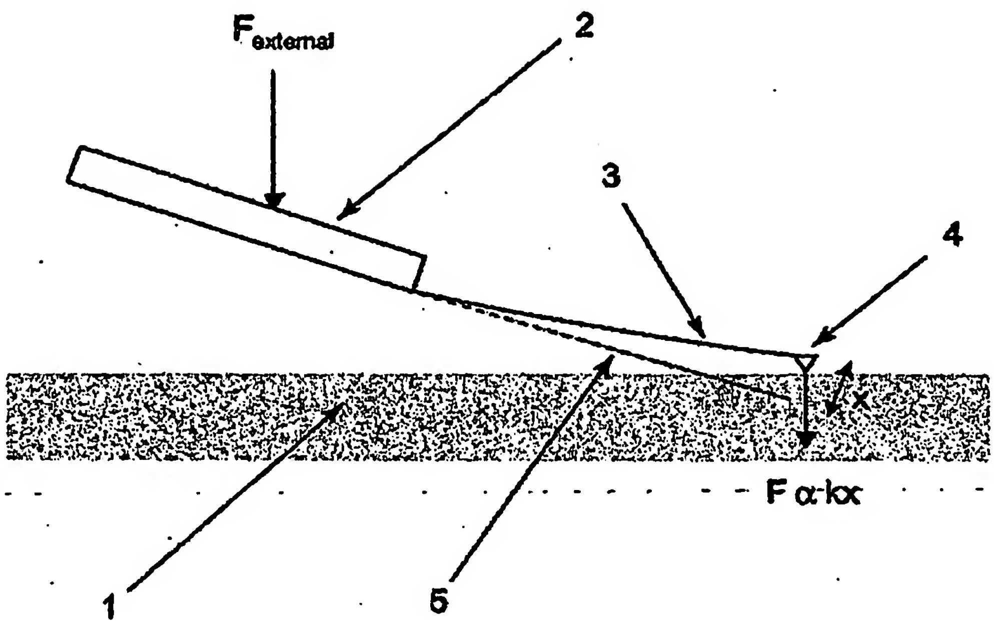
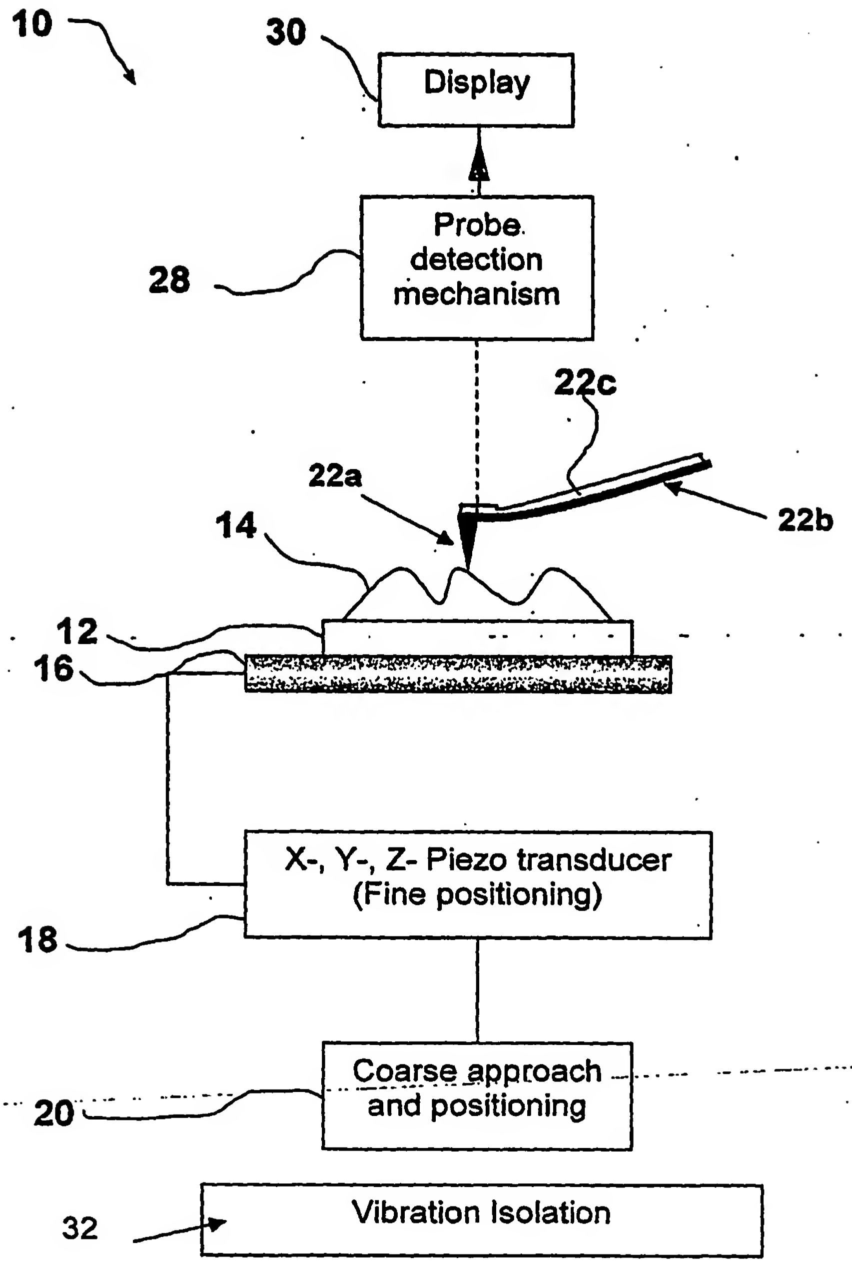


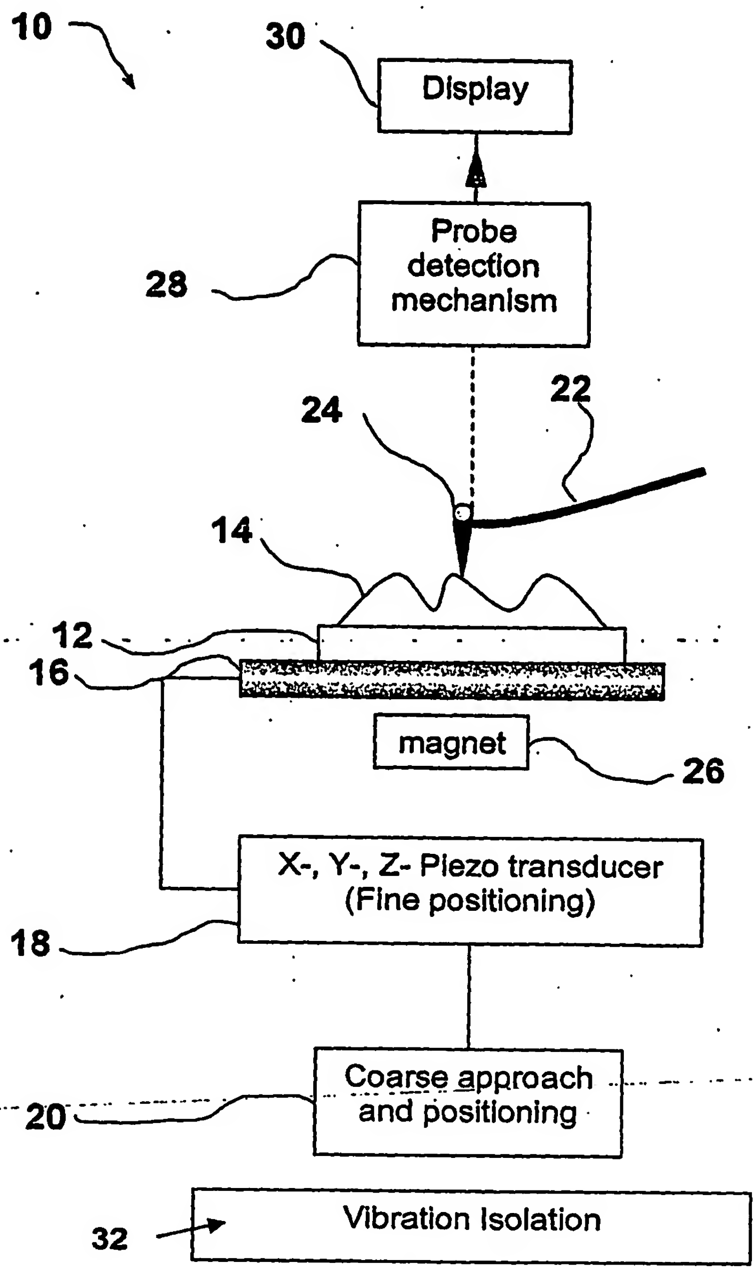
Fig 1

Prior Art



10
30
Display
Probe detection mechanism
28
22c
22a
22b
14
12
16
X-, Y-, Z- Piezo transducer (Fine positioning)
18
Coarse approach and positioning
20
Vibration Isolation
32

Fig 2



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Display
Probe
detection
mechanism
28
24
22
14
12
16
magnet
26
X-, Y-, Z- Piezo transducer
(Fine positioning)
18
Coarse approach
and positioning
20
Vibration Isolation
32

Fig 3

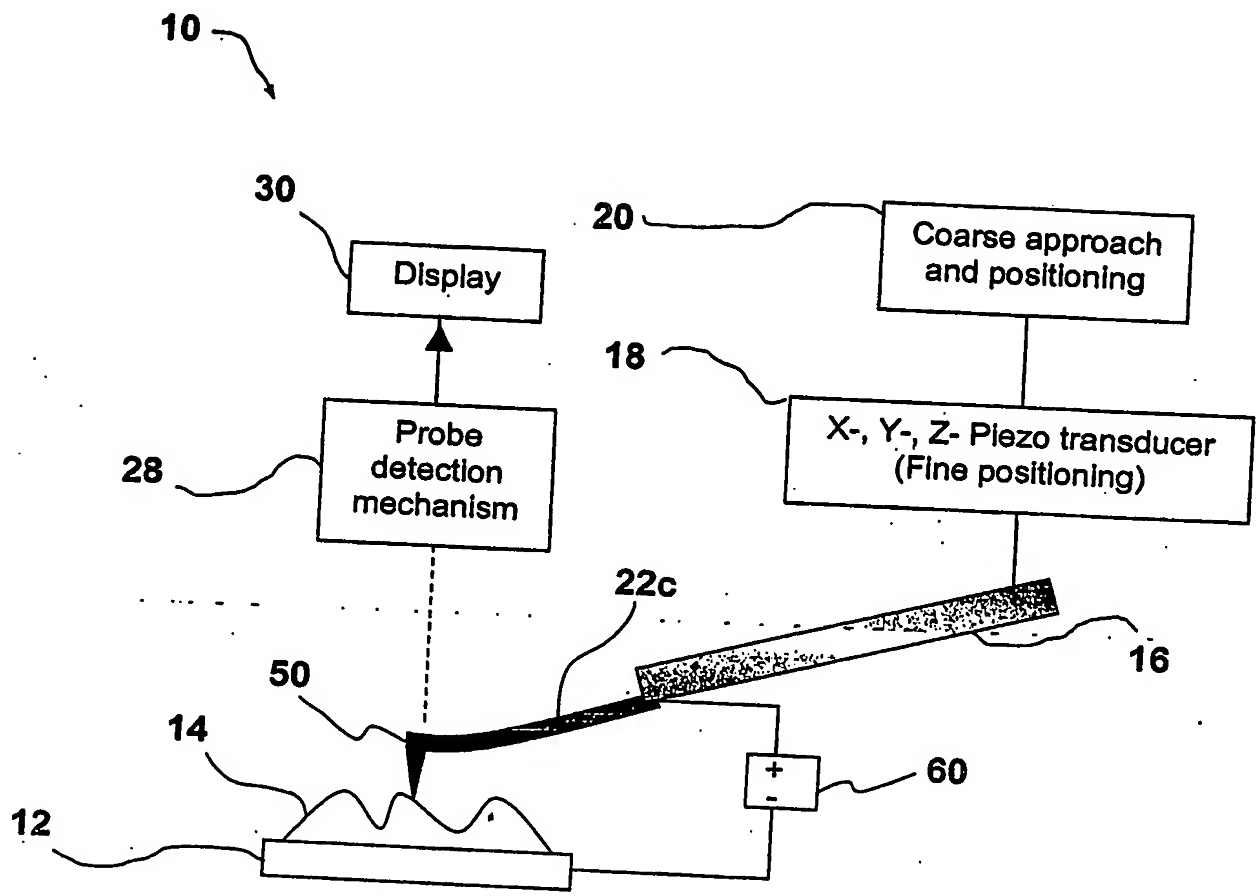

10
30
Display
20
Coarse approach and positioning
18
X-, Y-, Z- Piezo transducer (Fine positioning)
28
Probe detection mechanism
22c
16
50
14
12
60
+
-

Fig 4

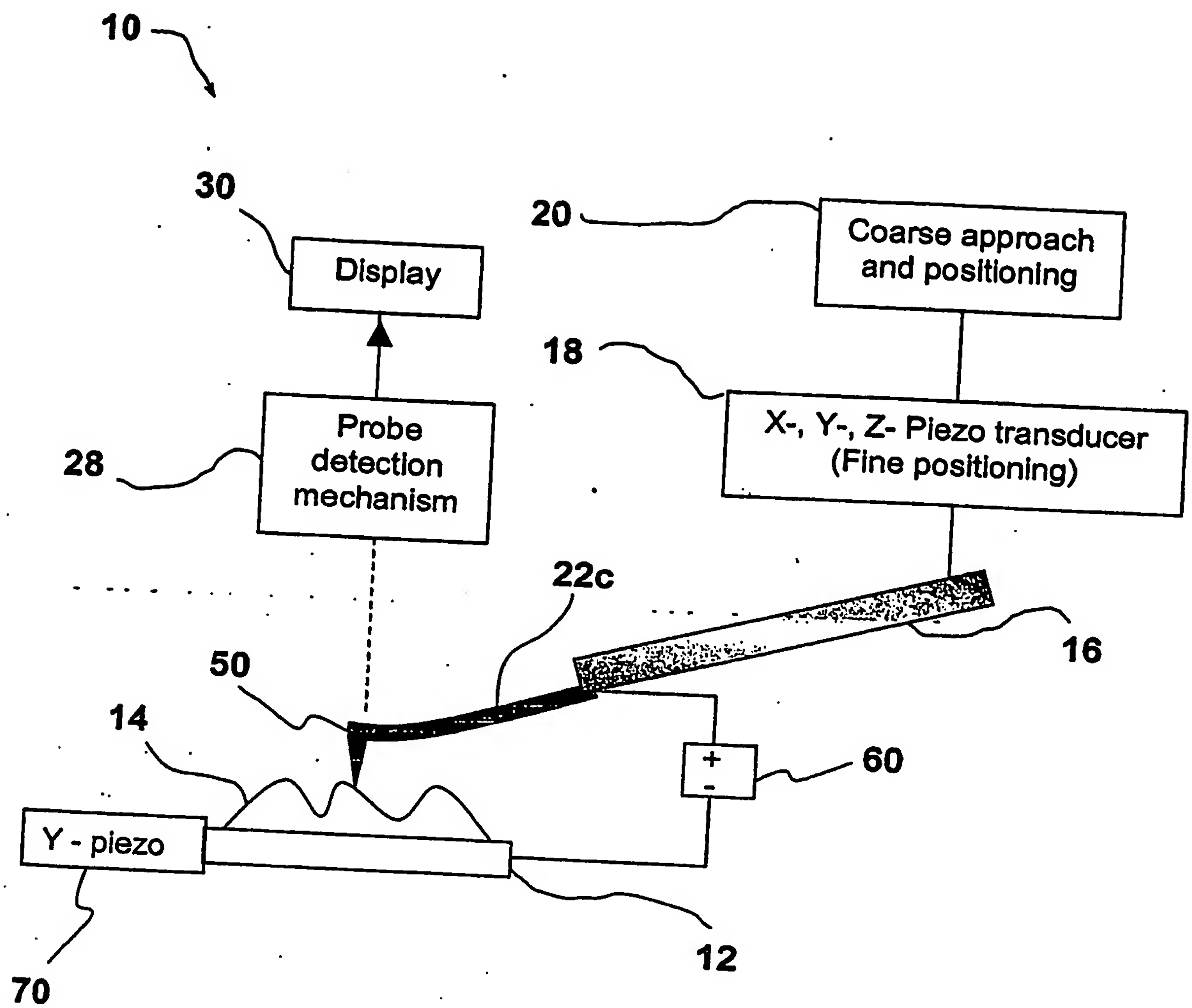
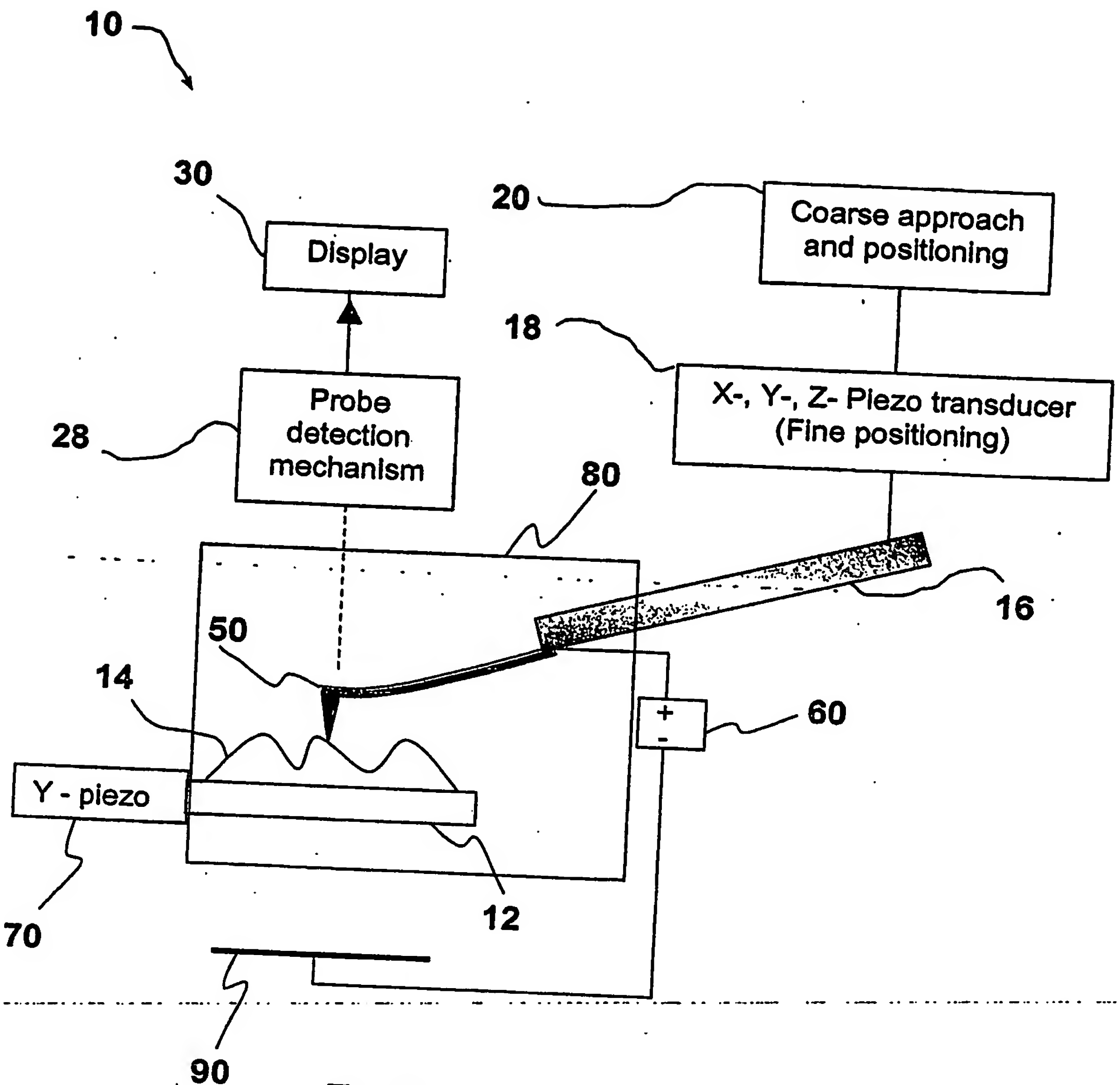
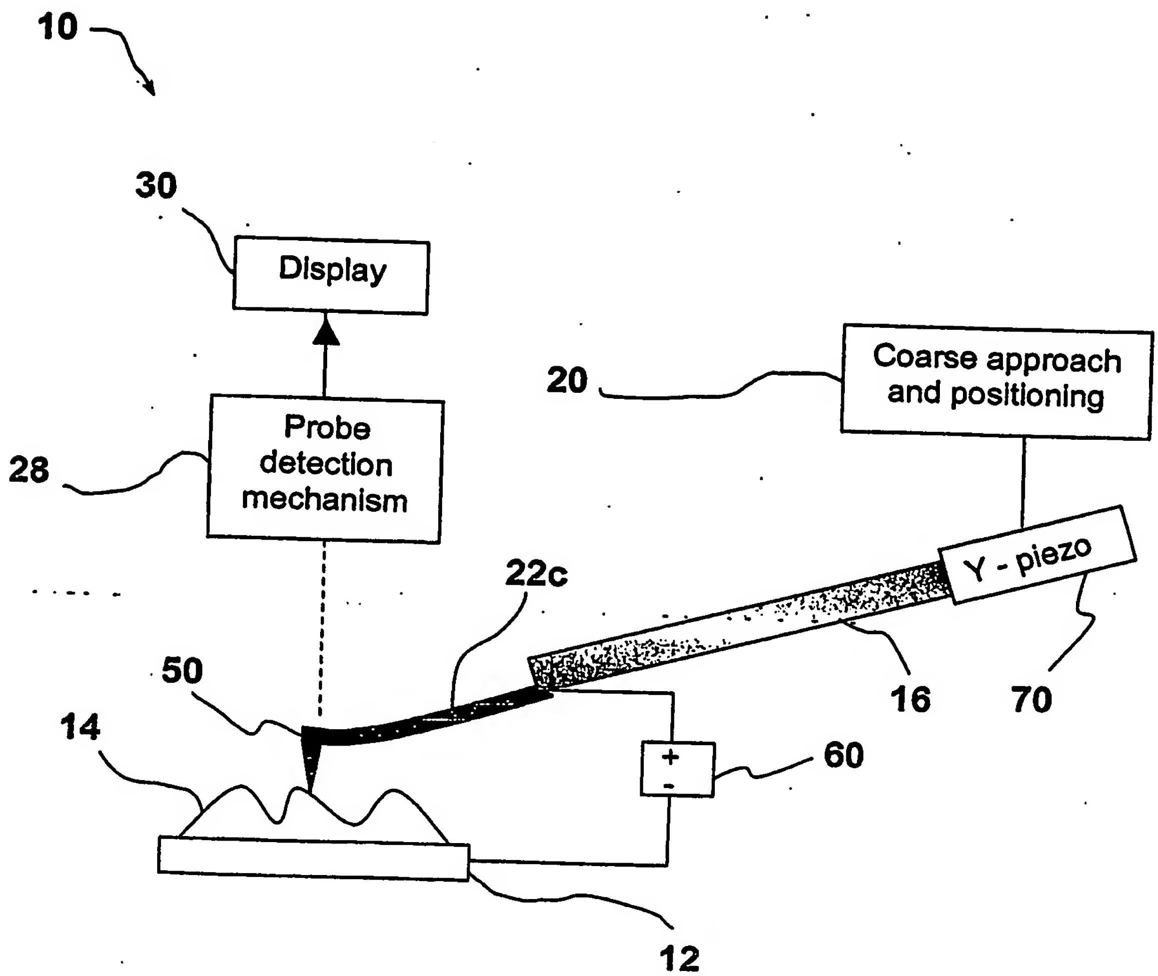

10
30
Display
20
Coarse approach and positioning
18
X-, Y-, Z- Piezo transducer (Fine positioning)
28
Probe detection mechanism
22c
16
50
14
60
+
-
Y - piezo
12
70

Fig 5



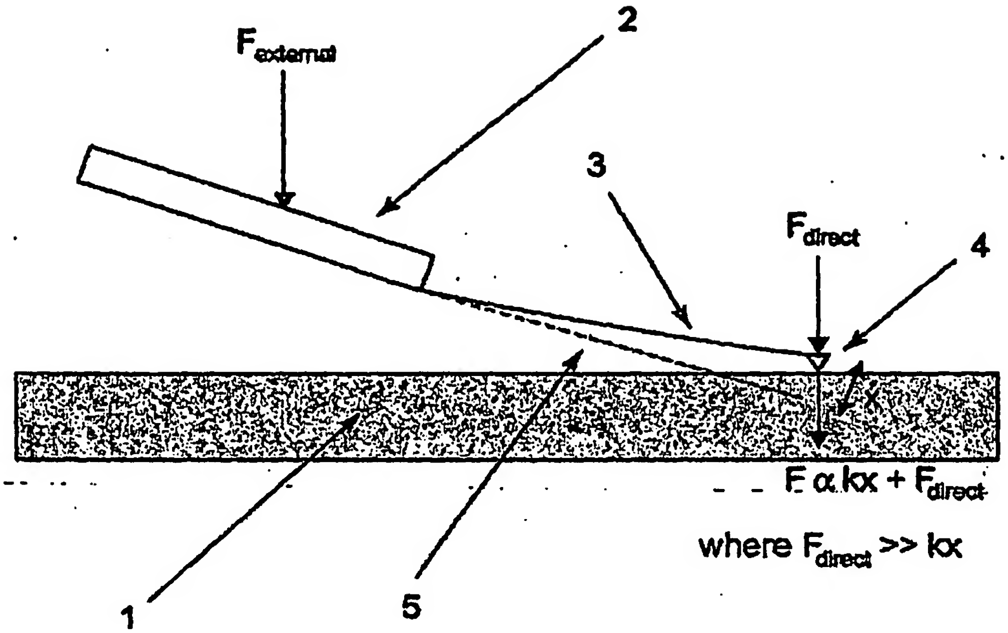
10
30
Display
20
Coarse approach
and positioning
18
X-, Y-, Z- Piezo transducer
(Fine positioning)
Probe
detection
mechanism
28
80
16
50
14
+
-
60
Y - piezo
70
12
90

Fig 6



10
30
Display
20
Coarse approach and positioning
28
Probe detection mechanism
22c
Y - piezo
50
14
16
70
+
-
60
12

Fig 7



$F_{external}$
2
3
F_{direct}
4
x
$F \alpha kx + F_{direct}$
where $F_{direct} >> kx$
1
5

Fig 8

Fig 9a Fig 9b Fig 9c

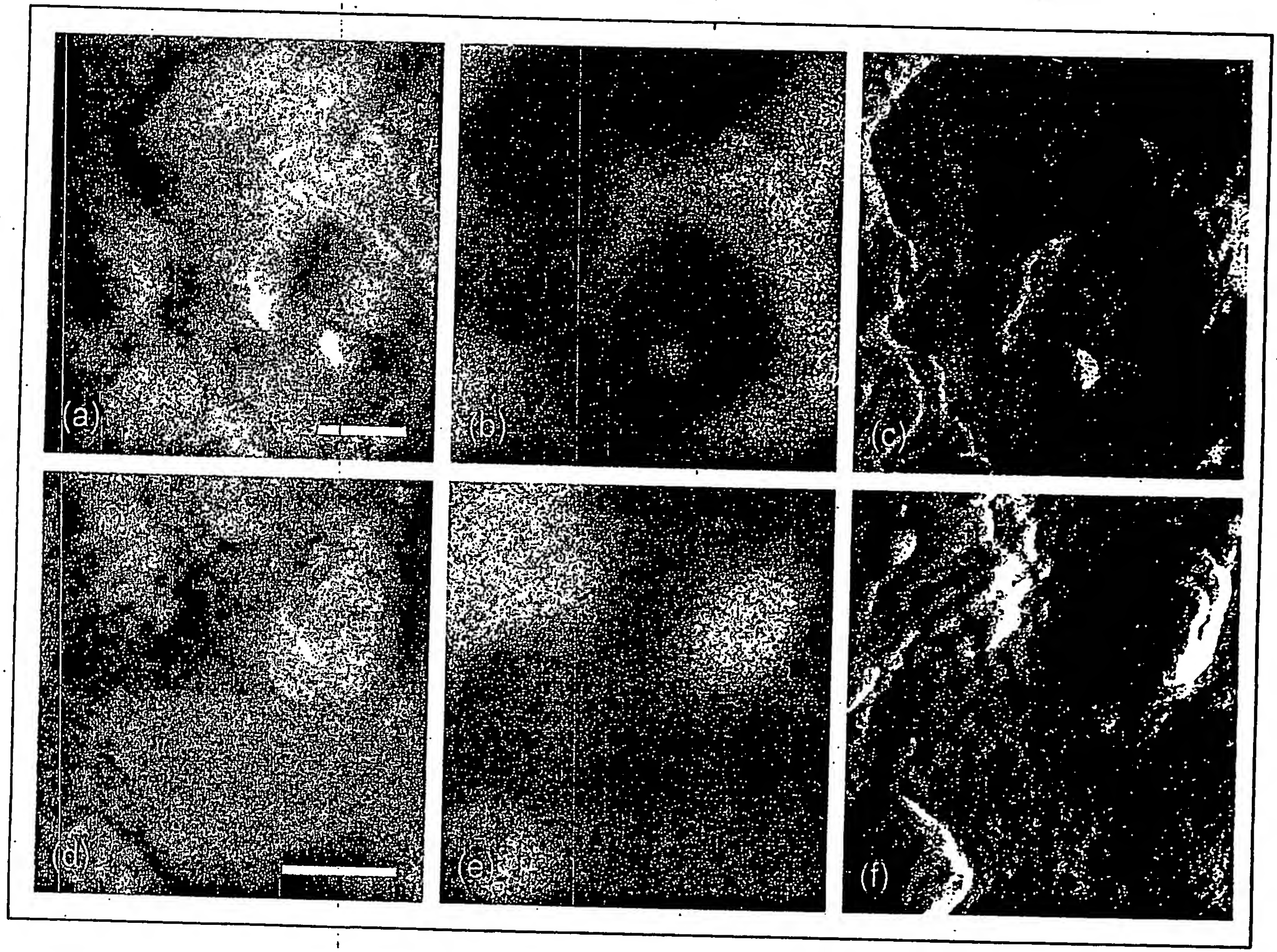


Fig 9d Fig 9e Fig 9f

PROBE FOR AN ATOMIC FORCE MICROSCOPE

This invention relates to the field of atomic force microscopes, to the probes employed in such microscopes and to a method of operating such microscopes. In particular, it relates to an atomic force microscope that does not employ conventional feedback control of probe height.

The atomic force microscope (AFM), or scanning force microscope (SFM), was invented in 1986 by Binnig, Quate and Gerber. Like all other scanning probe microscopes, the AFM is based on the principle of mechanically scanning a nanometric probe over a sample surface in order to acquire an "interaction map" of the sample. The interaction force in this case is simply the molecular interaction between the sample and the tip of a sharp probe attached to a cantilever spring. When the probe tip is brought into close proximity with the sample, the cantilever bends in response to the interaction force. Images are collected by scanning the sample relative to the probe and measuring the deflection of the cantilever as a function of lateral position. An optical lever technique is usually used to measure this bending. Since the cantilever obeys Hooke's Law for small displacements, the interaction force between the tip and the sample can be deduced.

The AFM is usually operated in one of two modes. In constant force mode, feedback enables a positioning piezoelectric driver to move the sample (or probe) up or down in response to any change in the interaction force that is detected. In this way, the interaction force may be held relatively steady and a fairly faithful topographical image of the sample is obtained. Alternatively the AFM may be operated in constant height mode. No, or very little, adjustment of the vertical height of the sample or probe is imparted during the scan. In this context, adjustment of the vertical height means that a translation is applied either to an actuator connected to the cantilevered probe or to the sample itself. There remains therefore a

degree of freedom for the probe tip to move up and down as the degree of cantilever bend is varied. In constant height mode, topographical changes to the sample are indistinguishable from interaction force variations in that either or both will cause the cantilever spring to bend.

In addition to these differing feedback regimes, image contrast is usually obtained in one of three different ways. In contact mode the tip and sample remain in close contact, i.e. in the repulsive regime of the molecular interaction, as scanning proceeds. In tapping mode an actuator drives the cantilever in a "tapping" motion at its resonant frequency. The probe tip therefore only contacts the surface for a very small fraction of its oscillation (tapping) period. This dramatically shortened contact time means that lateral forces on the sample are very much reduced and the probe is therefore less destructive to the specimen as the scan is taken. It is consequently much used for imaging sensitive biological specimens. Oscillation amplitude is generally held constant using a feedback mechanism. In non-contact operation the cantilever is oscillated above the sample at such a distance that the molecular interaction force is no longer repulsive. This mode of operation is however very difficult to implement in practice.

Recent advances in probe microscopy have led to much faster data collection times. With faster scan techniques, such as that described in PCT patent application publication number WO 02/063368, finite probe responsivity is increasingly becoming a limiting factor in image collection times. The probe will not respond instantaneously to a change in sample characteristics and so there is an inherent time delay between, for example, the probe encountering a region of the sample surface with increased height and the system reacting to it. This disadvantage applies to both constant force and constant height modes of AFM operation. It is less severe in constant height mode, which is therefore the preferred mode of operation for fast scanning techniques, but it is still sufficient to limit

unduly the scan speed of the current generation of fast scanning probe microscopes.

In constant force AFM mode, an electronic feedback mechanism is usually employed in order to keep the average interaction force constant. As the scan progresses if there is a change in interaction force (for example caused by a change in sample height) this is first observed by change in probe response which is detected by the detection electronics, an error is generated (e.g. set point minus deflection) and a feedback loop is used to minimise the error signal by adjusting the probe or sample position. The feedback loop has a time constant associated with it which imposes a limitation on the ultimate speed with which a full image scan can be collected.

The problem is not so restrictive if operating in constant height mode, in which electronic feedback is not normally employed to the extent that it is used in constant force AFM. For the interaction force to be measured accurately however the probe tip should, as far as possible, track the contours of the sample surface. This is ensured by exploiting the reaction force developed as the cantilever is bent by the sample surface. That is, as a high region of the sample surface is scanned, the cantilever is increasingly bent upwards and the energy stored in the spring is increased. As the height falls away, a restoring force pushes the cantilever back towards its equilibrium (straight) position, thus maintaining contact with the surface. If however the scan speed is too fast, the probe will not track the surface but will effectively be thrown upwards over any protuberance from the surface and may start to resonate, or "ring". This in turn gives rise to oscillations in the imaged interaction force. Similarly, when the height falls away the restoring force might not be sufficiently large to ensure that the probe tip remains in contact with the surface and information about the surface in that region of the image will be lost.

WO 02/063368, referred to above, describes a scanning probe microscope in which either the sample or the probe is mounted on a resonator and, by driving the resonator at or close to its resonant frequency, the sample can be scanned relative to the probe. The resonator will typically have a resonant frequency of several 10s of kHz, which is similar to the resonant frequency of the probe. The typical time spacing between pixels is therefore shorter than $1/f_r$, where f_r is the resonant frequency of the probe. On the other hand the time taken (τ_{res}) to respond to a change in topography of the sample surface is based on the effective mass of the probe and the spring constant of the cantilever. If $\tau_{res} > 1/f_r$, then clearly the interaction force will not be measured accurately from pixel to pixel.

There is a perceived need to provide for improved probe responsivity to sample topographic fluctuations or to variations in the interaction force and so to permit AFM microscopy to be performed at faster scanning speeds before image artefacts such as those caused by probe ringing or poor tracking of the surface start to degrade image quality.

The present invention provides a probe for use in an atomic force microscope or for nanolithography, the probe comprising a force sensing member connected to a probe tip having a tip radius of 100nm or less characterised in that the probe includes a biasing element which is responsive to an externally applied force for urging either or both of the probe tip and a sample towards each other. In an alternative aspect the present invention provides an atomic force microscope for imaging a sample in accordance with an interaction force between the sample and a probe, the microscope comprising

driving means arranged to provide relative scanning motion between the probe and the sample surface and capable of bringing the sample and probe into close proximity, sufficient for a detectable interaction to be established between them; and

a probe detection mechanism arranged to measure deflection and / or displacement of the probe;

characterised in that, the microscope includes the probe as described above.

Alternatively, the microscope is characterised in that, it includes force (F_{direct}) generating means arranged such that, in operation, a force (F_{direct}) is applied to either or both of the sample and the probe or between the sample and the probe, the force (F_{direct}) being directed so as to urge the probe towards the sample or *vice versa*.

In a further aspect the present invention provides a method of collecting image data from a scan area of a sample with nanometric features wherein the method comprises the steps of:-

(a) moving a probe having a supporting beam with a tip having a tip radius of 100nm or less into close proximity with a sample in order to allow an interaction force to be established between probe and sample;

(b) causing a force (F_{direct}) to be established between sample and probe such that the probe is urged to move towards the sample or *vice versa*;

(c) scanning either the probe across the surface of the sample or the sample beneath the probe whilst providing a relative motion between the probe and surface such that an arrangement of scan lines covers the scan area;

(d) measuring deflection and / or displacement of the probe; and

(e) processing measurements taken at step (d) in order to extract information relating to the nanometric structure of the sample.

With the present invention, unlike conventional AFM where image collection may take upwards of 30 seconds, millisecond imaging of samples is possible. For example, tip velocities of 22.4 cms^{-1} enables an area of 4.4 x 4.4 microns to be imaged in 14.3 ms and an area of 1.5 x 1.5 microns in 8.3 ms with 128 by 128 pixels. Moreover, even at this speed images with better than 10 nm lateral and 1 nm vertical resolution are achievable with a soft polymer surface.

Embodiments of the invention will now be described by way of example only and with reference to the accompanying drawings.

Figure 1 is a diagrammatic illustration of the forces involved as a cantilevered probe makes contact with a sample surface in a prior art atomic force microscope.

Figure 2 shows a schematic implementation of an atomic force microscope that includes a probe, which is in accordance with a first embodiment of the present invention.

Figure 3 shows a schematic implementation of an atomic force microscope that includes a probe, which is in accordance with a second embodiment of the present invention.

Figure 4 shows a schematic implementation of an atomic force microscope that includes a probe, which is in accordance with a third embodiment of the present invention.

Figure 5 shows a schematic implementation of an atomic force microscope that includes a probe, which is in accordance with a fourth embodiment of the present invention.

Figure 6 shows a schematic implementation of an atomic force microscope that includes a probe, which is in accordance with a fifth embodiment of the the

present invention.

Figure 7 shows a schematic implementation of an atomic force microscope that includes a probe, which is in accordance with a sixth embodiment of the present invention.

Figure 8 is a diagrammatic illustration of the forces involved as a probe makes contact with a sample surface in the AFM of Figures 2 to 7.

Figures 9a and 9d are AFM images of two separate surface regions of a sample of crystallised poly(ethylene-oxide) (PEO) produced using a probe in accordance with the present invention.

Figures 9b, 9c, 9e and 9f are conventional AFM images of the same surface regions as those of Figures 9a and 9d.

With reference to Figure 1, there is shown a sample 1 that is being scanned by a probe of an atomic force microscope (AFM). The probe comprises a substrate 2 from which a cantilever 3 extends, the cantilever 3 having a sharp probing nanometric tip 4 having a tip radius of 100nm or less, mounted at an end remote from the substrate 2. In preparation for a scan, a downwards force ($F_{external}$) is applied to the probe at its substrate end 2 via its mounting to the AFM, moving the probe tip 4 into contact with the sample 1. In order to maintain contact for the duration of a scan, the force $F_{external}$ is greater than that required simply to bring the tip 4 into contact with the sample 1. As a result the cantilever 3 is bent upwards from its rest position 5 as the sample is scanned.

The cantilever 3 obeys Hooke's Law for small displacements. Accordingly if, when pressing on the sample, the degree of bending is such as to move the tip 4 a perpendicular distance x from its rest position and the cantilever spring constant is k then the restoring force exerted by the cantilever is kx. The downward force exerted by the tip 4, holding it in position tracking the

surface, is thus proportional to kx.

Clearly the responsivity of the probe tip 4 and hence the resolution of the AFM technique depends on the degree of force kx exerted by the cantilever 3 on the sample 1. The greater the force between probe and surface, the greater the responsivity to surface variations. This indicates that a high spring constant k is desirable, particularly if the scan is to be fast. On the other hand, the greater the force, the more likely the probe is to damage the sample. Accordingly prior art AFM cantilever probes must make a fundamental compromise between probe responsivity and the likelihood of damaging the sample.

Figure 2 shows a schematic implementation of an AFM, indicated generally by 10, that utilises a first embodiment of a probe constructed in accordance with an aspect of the present invention. The AFM apparatus 10 shown comprises a plate 12 adapted to receive a sample 14, and which is mounted on one prong of a tuning fork 16. The tuning fork 16 is connected to a piezoelectric transducer 18 and a coarse driving means 20. The piezoelectric transducer 18 is used to drive the sample 14 (together with the plate 12 and fork 16) in three dimensions: x, y and z directions. As is conventional in the field, the z axis of a Cartesian coordinate system will be taken to be that perpendicular to a plane occupied by the sample 14. That is, the interaction force is dependent both on the xy position of a probe 22 over the sample 14 (the pixel it is imaging), and also on its height above it. A tuning fork control (not shown) is arranged to apply a sinusoidal voltage to the tuning fork 16 and so to excite a resonant or near-resonant vibration within the xy plane. Optionally, the plate 12 and tuning fork 16 may be supported on a vibration isolating mount 32 so as to isolate the vibration of the tuning fork 16 from the remainder of the microscope. However, at the image frequencies contemplated with a microscope employing this probe, external noise is less of a problem than for lower image frequencies and so the vibration isolating mount may be dispensed with. The probe 22 is a

low-mass AFM probe and, during a scan, an interaction force is developed between the probe tip 22a and the sample surface. A probe detection mechanism 28 is arranged to measure the displacement of the probe tip 22a or the bending of the beam 22b supporting the tip, which is indicative of interaction force strength. Data collected by the probe detection mechanism 28 is analysed and output to a display 30.

In general, prior art cantilever probes are fabricated from silicon or silicon nitride, which allows them to be produced readily using mature silicon microfabrication technology. Unlike prior art cantilever probes however, the probe 22 according to this invention has a polymer coating 22c applied to the supporting beam 22b of the probe. This coating 22c, as will be explained in more detail later, serves to dissipate energy that would otherwise be mechanically stored in the probe through the excitation of oscillatory modes and thereby lowers the Q factor of the supporting beam for one or more of its vibrating modes in comparison to the same beam without the presence of the coating 22c.

In taking images using the apparatus 10, the sample 14 is first brought into contact with the probe 22 using the coarse driving means 20. Fine height and initial start position adjustments are made with the piezo driver 18 whilst the probe detection mechanism 28 measures the bending of the probe as a result of the probe 22 – sample 14 interaction force. Once the measured bending reaches a desired level, the sample surface is scanned beneath the probe 22. In scanning the sample 14 under the probe 22, the tuning fork 16 is set to vibrate into and out of the plane of the Figure (*y* axis). This oscillates the stage on which the sample is mounted. At the same time, the piezo 18 translates the sample 14 in a perpendicular (*x*) direction. Sample oscillation is with a relatively large amplitude, of the order of a few microns. During the course of a scan, readings are continually taken by the probe detection mechanism 28, which, as is standard in the art, may be based on an optical lever technique: probe

bend is measured using laser light reflected from the probe. The output signal from the probe detection mechanism 28 is fed directly to a processor and display 30.

As stated above, the probe 22 shown in Figure 2 differs from those of the prior art in that it is coated with a polymer material 22c. The coating 22c may be on one or both sides provided that the material itself is suitable for dissipating energy that would otherwise be stored in the probe.

The Q factor is a dimensionless quantity, which may be used to quantify the dissipation (or damping) of an oscillator. It has the property that:

$$Q = \frac{\text{Energy stored in oscillator}}{\text{Energy dissipated per radian}}$$

A heavily damped system, in which stored energy is dissipated rapidly, has a low Q, and a lightly damped system has high Q. Oscillators made from Si and SiN materials do not have much internal loss and, as a result, most commercially available AFM cantilevers will have high Q, typically of the order 50 – 500 in air. Moreover, if designed for use in tapping mode, it is advantageous for a cantilever to have a high Q. In this mode, the cantilever is driven at resonance and the interaction force measured over many cycles of oscillation. By minimising energy loss over the oscillation cycles, the high Q therefore acts as a mechanical filter.

A mechanical oscillator has many resonant modes of oscillation and the quality factor of each of these modes can be different, depending on the frequency dependent material properties and the shape of the oscillator. When referring herein to the Q factor we are referring to the Q factor of the probe with respect to any one of these modes, or to the Q factors of a set of modes

In the case of the present invention however, it is desirable to use a probe

with low Q in high-speed atomic force microscopy. If the probe has a high Q, it will take a long time to respond to changes and it will ring at a combination of resonant modes if given a stimulus, such as provided by scanning across a high feature on the sample surface. The present probe is designed to have a low Q by virtue of its coating 22c. The Q factor is, ideally, sufficiently low such that any induced oscillation is critically damped. The use of a low quality factor means that little energy can be stored in the supporting beam of the probe and so the probe will not "ring" for long if shocked, such as when scanning over a high region of the sample surface. This enables a speedier return to the sample surface, and consequently its better tracking during a scan.

The coating on the probe acts to dissipate mechanical energy that would otherwise be stored in the probe. The probe with the coating will store less mechanical energy than the probe without the coating, and the motion of the probe with the coating at a specific time will relate more closely to the surface under the probe tip at that specific time, than it would do if the coating was not present.

Depending on the sample being imaged and the scan-speed chosen, it may be that a higher mode than the first or fundamental mode is most likely to be excited during imaging. In this case the coating is chosen to ensure that the Q factor of this mode is significantly reduced. By tuning the energy absorbing and dissipating properties of the coating it is possible to reduce or remove oscillations of the probe that are most likely to interfere with image quality while minimising the change in mass of the probe.

Many polymer materials may be used to provide the coating 22c, and the opportunities for specific selection will be apparent to one skilled in the art. A block copolymer material in which the majority component is an amorphous rubber, with glass transition temperature below room temperature, and the minority component is an amorphous polymer with a

glass transition temperature above room temperature, coated on both sides of an AFM supporting beam has been found to improve markedly its tracking capability when used at room temperature. The copolymer was applied by solution casting. That is, a drop of solution containing the polymer is placed on the supporting beam at high temperature in order to drive off the solvent. Other thermoplastic elastomers may also be used. Such an arrangement has been found to permit the probe to track a sample surface even at resonant oscillation speeds such as described in WO 02/063368.

Considerations as to the polymer material and application method adopted narrow the available choice to some extent. The basic idea is to coat the supporting beam with an energy-absorbing material that, ideally, does not unduly affect other properties of the probe such as mass, sharpness of tip, etc. Solution casting the supporting beam with the above-described copolymer has been found to enhance energy dissipation with an acceptable increase in mass. Other coating methods can be used however. These include: "dragging" a charged polymer onto the supporting beam in an electrolysis cell; chemically tagging the polymer (for instance with a thiol group) and using its reaction with the material of the supporting beam, or metal coating on the supporting beam (e.g. gold in the case of thiol chemistry), to attach the polymer to the supporting beam.

Applying a coating 22c to both sides of the supporting beam, given its small size, is, practically, somewhat easier to achieve than coating one side only. It is however preferred that the side of the supporting beam nearer to the sample is left uncoated. The single-sided coating is sufficient to reduce the mechanical energy stored in the probe and also reduces the likelihood of any coating material contaminating the sample when the probe makes contact.

Ideally the polymer material used for the coating 22c will have a peak in its

energy loss spectrum at the temperature of the probe's anticipated use and in the frequency range of the principal resonant modes of the supporting beam. Typically, it should therefore be a rubbery polymer. Alternatively a copolymer or other composite with a high component of rubbery polymer may also be used.

Figure 3 shows a schematic implementation of an AFM, indicated generally by 10, that utilises a second embodiment of a probe constructed in accordance with the present invention. The AFM apparatus 10 is very similar to that shown in Figure 2, and components common to both systems are similarly referenced. As before, the plate 12 holding the sample 14 is mounted on one prong of the tuning fork 16, which is driven with a resonant or near-resonant vibration within the *xy* plane. The sample 14 (together with the plate 12 and fork 16) is scanned in three dimensions: *x*, *y* and *z* directions, with the interaction force developed being dependent both on the *xy* position of the probe 22 over the sample 14 (the pixel it is imaging), and also on its height above it. The cantilever component of the probe 22 is coated on both sides with a polymeric film and is shaped so as to have a low spring constant, less than 1 Nm^{-1}. Unlike the cantilever shown in Figure 2 however, the probe 22 according to this embodiment of the invention additionally has a magnetic element 24 (a bead is illustrated in Figure 2) mounted above the tip 22a. Also a magnet 26 is incorporated within the AFM, for example below the plate 12, to provide a magnetic field of sufficient strength to exert a force on the magnetic bead 24. The probe detection mechanism 28 is arranged to measure the bending of the probe 22, as for the apparatus 10 shown in Figure 2. Data collected by the probe detection mechanism 28 is analysed and output to a display 30.

In taking images using the apparatus 10, the contact mechanism to establish an interaction force and scanning technique are substantially as described in relation to the apparatus 10 of Figure 2. Once the desired level of interaction force, and hence bend of the supporting beam 22b of

the probe, is established however, then the magnet 26, which is not present in the Figure 2 apparatus 10, is switched on and a magnetic field *B* is generated in the vicinity of the probe tip 22a. The magnetic bead 24 interacts with this field, which is directed such that the resultant magnetic force attracts the magnetic bead 24 downwards towards the sample 14. The probe tip 22a is therefore held in contact with the sample 14 by the direct action of this magnetic force. With the magnetic field *B*´on, the sample surface is oscillated (at the resonant frequency of the tuning fork-sample stage) and scanned beneath the probe 22 and the output signal processed as before.

Figures 4 through 7 show schematic implementations of alternative AFMs, indicated generally by 10, that utilise further embodiments of a probe constructed in accordance with the present invention. In each case the AFM apparatus 10 is very similar to that shown in Figures 2 and 3, and components common to all apparatus are similarly referenced. As before, the sample 14 is mounted on a plate 12. Dfferent from the embodiments illustrated in Figures 2 and 3, in Figures 4, 5, 6 and 7 the probe 22 is mounted on one prong of the tuning fork 16 and control of the approach and both coarse and fine positioning of the probe relative to the sample is controlled by transducers18, 20, such as piezo transducers, which control movement of the probe 22 and tuning fork 16 rather than the plate 12. This arrangement allows the probe to be scanned using the resonant scanning method above the sample, rather than the sample being scanned below the stationary probe. With respect to Figure 4 the resonator 16 and probe 22 are scanned in the x-axis using the x-y-z piezo transducer 18 while in Figures 5 and 6 control of relative probe / sample movement in the scan direction (x direction) during a scan is provided by means of a transducer 70 connected to the plate 12. Thus the sample can either be scanned in both axes while the probe is stationary, or the probe can be scanned in both axes while the sample is stationary, or one or other of the probe or sample can be scanned in one axis, while scanning in the other axis is

provided by motion of the other. In the case of Figure 7 control of the relative probe / sample scan movement is provided by a transducer 70 connected to the resonator 16 and probe 22 and fine positional control is omitted as such precision of the starting scan position is not in all cases required. This highlights an additional advantage of the very fast scan rates that are obtainable using the described invention in conjunction with the resonating scanning method. The image rate is higher than common frequencies for mechanical noise, and higher than the instabilities in motion that are commonly present in course positioning methods. Thus it is possible to dispense with the high precision piezoelectric transducers that are usually required.

In Figures 4, 5 and 7 the probe tip 22a is subjected to a force urging the tip 22a towards the sample 14. In the example of Figure 4 the force is attractive and arises from a biasing voltage being applied between the probe tip 22a and the plate 12. Hence, the probe tip 22a and the plate 12 are connected in series across the terminals of a power supply 60. In order to establish the necessary attractive force between the probe tip 22a and the plate 12, the probe is provided with an electrically conductive coating 50 in addition to the damping coating 22c to ensure the probe has a low Q factor. In the case of Figure 6 the sample 14 and probe tip 22a are positioned within a sealed viscous environment 80, such as a liquid environment. In this embodiment the power supply 60 is connected across the conductive coating 50 and a second plate 90 located beneath the sample plate 12 outside of the viscous environment. By immersing the probe in a liquid (which in the case of biological samples may be desirable) the damping coating 22c may be omitted from the probe as the exposure of the probe to the liquid environment results in the probe having a low Q factor approximating 1.

In order to appreciate the features that are necessary to this invention it is helpful to look at a diagrammatic representation of the forces involved while

a scan is being performed. This is illustrated in Figure 8, which shows the same set up as Figure 1 and so like components are similarly referenced. With reference to Figure 8, there is shown a sample 1 that is being scanned by a probe of an atomic force microscope (AFM) in accordance with the present invention. The probe comprises a substrate 2 from which a supporting beam 3 extends, the supporting beam 3 having a sharp probing tip 4 mounted at an end remote from the substrate 2. In preparation for a scan, a downwards force ($F_{external}$) is applied to the probe at its substrate end 2 via its mounting to the AFM, moving the probe tip 4 into contact with the sample 1. In order to maintain contact for the duration of a scan, the force $F_{external}$ is greater than that required simply to bring the tip 4 into contact with the sample 1. As a result the supporting beam 3 is bent upwards from its rest position with F_{direct} present, 5, as the sample is scanned. As before, a force proportional to kx is generated as a result of the supporting beam bending and directs the probe tip 4 downwards towards the sample surface.

In the event that a probe designed in accordance with the present invention is deflected from the sample surface, for example by encounter with a raised portion, two factors assist in restoring it back towards contact. This enables better tracking of the surface to be achieved, even at high scan speeds. First, as is seen most clearly in the embodiments shown in Figures 3-7, a second force F_{direct} acting to accelerate the probe towards the sample, can be tuned so as to reduce to a minimum the time it takes to bring the probe back into contact with the surface. This force, which is largely independent of topography, acts to reduce the response time of the probe. Secondly, the probe is coated with an energy absorbing material which reduces the mechanical energy stored in the probe and so reduces the effect that previous impulses have on its motion, ensuring that it rapidly obtains a stable state in contact with the surface.

The total restoring force holding the probe to the surface is now

proportional to:

$$F_{direct} + kx,$$

Ideally, the additional force F_{direct} is greater than the cantilever bending force kx. Its magnitude should moreover be sufficiently large to bring the probe into contact with the surface, should it lose contact, within approximately one pixel.

In the embodiment depicted in Figure 3, the additional force F_{direct} is a magnetic force, provided by applying a magnetic field to a probe tip that incorporates a magnetic element such as a bead or a magnetic coating. Clearly therefore the positioning of the magnet within the AFM is not critical, it merely has to be arranged such that there is a downward force component pulling the probe tip 4 into the sample 1. In the subsequent embodiments the additional force F_{direct} is an electrostatic force.

In the embodiment depicted in Figure 2, the additional force F_{direct} is still contributing to the tracking performance of the probe, but its origin is more subtle. As the probe and sample are brought into close proximity a capillary neck is generally believed to form, connecting the two. This capillary neck is thought to arise from fluid that will inevitably be present in the sample environment when it is imaged in air, which condenses about the probe – sample contact. In normal operation, it is found that the direct force F_{direct} arising from the capillary neck is sufficiently large that it quickly forms the dominant restoring force on the low-Q probe i.e. $F_{direct} > kx$. This is particularly true for hydrophilic surfaces. By choosing a probe that has a hydrophilic surface, for instance silicon nitride, it is possible to ensure that a capillary neck is formed between the probe and the sample.

Regardless of the origin of the additional direct force F_{direct}, the low Q of the probe permits stored energy to be dissipated rapidly as the supporting

beam is straightened and the probe's contact with the sample surface is restored by the action of the direct force F_{direct}. Tracking of the sample surface by the probe is therefore achieved by a kind of mechanical feedback loop, which is faster acting than the prior art tracking mechanisms with their dependency on the cantilever bending force kx.

In the microscope described herein, the end of the probe is responding at a frequency considerably higher than its first mode of oscillation. Therefore, there is no longer a simple relationship between the bending of the probe, and its vertical position, as the degree of bending will now depend on how long it has been at that vertical position. Therefore, images obtained using a method based on the reflection of a laser from the back of the probe onto a split photodiode will not correspond to the topography of the surface, but rather to a combination of the topography and the gradient. To obtain images that do correspond to topography, the displacement of the probe can be monitored for example using an interferometric method . For instance, a fibre interferometer may be used to monitor the position of the end of the probe relative to the fibre, or an interferometer based on a Wollaston prism may be used to monitor the position of the end of the probe relative to another point, or an interference microscope may be used to monitor the position of the end of the probe, in which case the optical intensity at a position in the field of view of the microscope that corresponds with the end of the probe will vary depending on its vertical position. Whichever method is used, an image can now be obtained that will correspond to the topography of the surface, with particular application for metrology.

In order to assist in achieving $F_{direct} > kx$, the probe should be further designed with a relatively low spring constant. Typically this should be less than 1 Nm^{-1}, which can be achieved by using a suitably shaped probe. In

the present invention, the cantilever force kx is useful only to define the position in space at which the probe sits, i.e. the interaction force between probe and sample, and so to enable an image to be collected.

The ability to exploit a direct restoring force F_{direct} as opposed to relying on the cantilever force in sample tracking represents a significant improvement over the prior art. By providing a probe that has a reduced ability to store mechanical energy, the principal forces acting on the probe are the direct force F_{direct}, and the force due to the immediate bending of the probe by the surface, with the direct force F_{direct} being the dominant force. This applies regardless of whether the direct force is a "natural" force, generated by means of the capillary neck, or an additional, external force, such as that applied via a magnetic bead. In either case, the restoring force has a magnitude that is substantially independent of the position of the probe. By way of contrast, the magnitude of the prior art restoring force kx depends on the displacement x of the cantilever from its rest position. Thus high restoring forces are generated at particularly high regions of the sample. It is very difficult to ensure consistently that samples are not damaged if the restoring force is permitted to vary in this manner. A restoring mechanism implemented in accordance with this invention has a magnitude that is largely independent of sample height.

As illustrated, it is not essential that the applied force is a magnetic force, although it is preferred that it is a force whose magnitude does not depend on sample height. It is required that there is a net force towards the surface so that any force from oscillatory modes present in the probe does not cause the probe to leave the surface. The larger the direct force F_{direct} therefore, the less strict is expected to be the requirement for energy absorption and dissipation by the coating.

Figures 9a to 9f clearly illustrate the improvement in performance of the probe of the present invention over conventional AFM apparatus. Figures

9a, 9b and 9c are all images of the same surface region and Figures 9d, 9e and 9f are similarly all images of another surface region. In all cases the scale bar represents 1 micron and the material of the surface being imaged was crystallised poly(ethylene-oxide) (PEO) mounted on a glass substrate. Figures 9a and 9d are images produced using the probe of the present invention whereas Figures 9b and 9e are images produced using a conventional AFM monitoring changes in probe height and Figures 9c and 9f are images produced using a conventional AFM monitoring deflection changes. To produce the images of Figures 9a and 9d a Veeco Dimension 3100™ AFM with Nanoscope™ IV controller was used with commercially available cantilevers coated in a thin polymer film. The sample was mounted on a micro resonant scanner constructed from a quartz crystal resonator and 5 micron piezo stack (P-802 and E-505, Physik Instrument, Germany). To collect the data in Figures 9a and 9d the Resonant Scanning Controller of Infinitesima Ltd was used.

Figures 9a and 9d were constructed from a 128 x 128 pixel array over a period of only 14.3 ms, the probe tip velocity in the centre of the image being 22.4cms^{-1} and 16.8cms^{-1} respectively.

Thus with the present invention images of areas of a few microns can be produced in milliseconds unlike conventional AFM where image collection may take upwards of 30 seconds. Although the illustrated embodiments can be operated with scanning tip velocities equivalent to those currently employed with conventional AFM microscopy, the embodiments are capable of tip velocities upwards of 0.1 cms^{-1} and depending upon the eveness of the sample surface tip speeds in excess of 50.0 cms^{-1} can be achieved. For example, with a tip velocity of 22.4 cms^{-1} an area of 4.4 x 4.4 microns can be imaged in 14.3 ms and an area of 1.5 x 1.5 microns in 8.3 ms. Moreover, even at this speed images with better than 10 nm lateral and 1 nm vertical resolution are achievable with a soft polymer surface.

Furthermore, observations suggest that at these probe tip velocities the sample is subjected to less damage than at lower speeds. As the probe tip spends less time at each point, the sample is subjected to less deformation and is therefore less likely to reach a point where it starts to deform plastically. With the present invention the surface of the sample can be subjected to shear rates of around 10^8 ms^{-1} which is a rate at which many polymers, for example, exhibit glass characteristics. In general, it has been found that higher frequencies can push a visco-elastic liquid down through the glass transition temperature and therefore change the properties of the surface that the probe 'sees' resulting in less damage to the sample.

The probe of the present invention is selected to have a low Q, ideally such that any induced oscillation is critically damped. As described herein, the most preferred arrangement, and one which is sufficiently effective to enable improved tracking by means of the natural restoring force due to the capillary neck, is to coat one or both sides of the supporting beam of the probe with an energy absorbing material, such as a polymer film. An alternative, particularly if a large magnetic (or other additional) force is applied, means to ensure low Q is by judicious selection of probe shape. Another alternative is simply to provide a low Q factor by immersing the probe in a viscous / liquid environment during the scan or electronically altering the properties of the supporting beam of the probe for example where the supporting beam is formed from or includes electro-responsive materials which can be addressed to provide a lower effective Q factor.

The supporting beam, probe tip and any additional component such as the magnetic bead are ideally of low mass. This naturally increases the acceleration of the tip back to the surface for a given restoring force and so better enables the probe to track the surface.

It is to be noted that the apparatus shown in Figures 2 through 7 are merely illustrative of exemplary AFMs. There are numerous different embodiments

of AFM with which this invention may be implemented all of which omit conventional feedback control of probe height as the principle method for obtaining an image. For example, mounting on a resonator such as a tuning fork is not necessary. This arrangement is simply used in these embodiments in order to illustrate the applicability of this invention to fast scanning techniques that make use of a resonant oscillation. It is equally applicable to slower scanning methods. The probe 22 may alternatively be oscillated in place of the sample 14. With this alternative embodiment it is envisaged that where optical techniques are employed to monitor the displacement of the probe, the imaging beam is broad enough to encompass the fast scan axis.

Probe deflection / displacement may be measured by means other than the optical lever technique. Alternative techniques known in the art include interferometry and piezoelectrically coated probes as well as detection of thermal variations in the radiant output of a heated probe. By employing interferometry for monitoring the deflection / displacement of the probe it is possible to extract purely topographic data of the sample surface from probe deflection data which, because of the frequencies at which the probe is responding, is representative of both the topology of the sample surface and gradient. Also, although the use of piezoelectric actuators for control of the movement of the sample plate / probe are preferred, other actuators involving for example thermal expansion of a control rod, are envisaged.

Although control of the Q factor of the probe has been described in terms of providing an energy absorbing coating to the supporting beam of the probe, other means for controlling the Q factor of the probe are envisaged including electronic control.

In order to image surface areas larger than the scan area of the probe, separate sequential images of different, usually adjacent, regions can be generated and then combined to construct an image over that larger area.

Stepper motors or other actuators may be used to move the probe and / or sample plate between the separate images before the fine positional adjustment for each individual image. Ideally the individual scan areas are selected to overlap so that visual confirmation of alignment of the individual images is possible.

If a tuning fork 16 is used then it may be one of a number of commercially available forks, or of bespoke design to provide a desired frequency of oscillation. A suitable example is a quartz crystal fork with resonant frequency of 32 kHz. A tuning fork is well suited to this application as it is designed with highly anisotropic mechanical properties. Its resonances are therefore independent and can be individually excited and so limited to only that (or those) in the plane of the sample. Importantly, the fork 16 can be resonated in one direction and scanned in another, without coupling occurring between modes. It therefore permits stable fast motion of the sample 14 as it is interrogated by the probe 22. Alternative mechanical resonators that have a similar facility for well-separated lateral and vertical resonances can be used in place of the tuning fork.

The invention is not limited to pure AFM operation, although it is required that there is a force interaction between the probe and the sample surface. This mode of operation can however be combined with microscope components designed to monitor other interactions or interaction indicators between probe and sample. Examples of other interactions may include optical, capacitative, magnetic, shear force or thermal interactions. Other indicators include oscillation amplitude, either tapping or shear force, capacitance or induced electric currents. These various modes of operation of general probe microscopes are described, for example, in UK patent application number 0310344.7.

The interaction of the probe with the sample surface that is exploited in AFM also makes it possible to affect the properties of the surface and so

deliberately to "write" information to the sample. This technique is known as nanolithography, and AFMs are widely used for this purpose. For example, by application of a voltage to a conductive cantilever a region of a metallic layer of a sample wafer can be oxidised. Another example exploiting two-photon absorption and polymerisation of a photoresist is described in "Near-field two-photon nanolithography using an apertureless optical probe" by Xiaobo Yin *et al.* in Appl. Phys. Lett. 81(19) 3663 (2002). In both examples the very small size of the probe enables information to be written to an extremely high density. The AFM and cantilever probe of this invention can also be adapted for use in nanolithography. The ability to improve surface tracking with this invention not only offers the potential for faster writing times than previously achieved, but also offers the potential for increased image resolution i.e. write density. To render it more adapted for use in nanolithography the probe tip may be electrically conductive, it may be metal coated in order to increase its optical interaction with the surface or it may be coated with selected molecular species for use in dip pen lithography applications.

CLAIMS

1. A probe (22) for use in an atomic force microscope or for nanolithography, the probe comprising a force sensing member (3) connected to a probe tip (4) having a tip radius of 100nm or less characterised in that the probe includes a biasing element (24, 50) which is responsive to an externally applied force for urging either or both of the probe tip (4) and a sample towards each other.

2. A probe (22) as claimed in claim 1 characterised in that the biasing element comprises a magnetic element (24) responsive to an externally applied magnetic force.

3. A probe as clamied in claim 2 characterised in that the magnetic element (24) is mounted on the force sensing member (3) adjacent the tip (4).

4. A probe (22) as claimed in claim 1 characterised in that the biasing element comprises an electrically conductive member (50) adapted for connection to one terminal of a power supply (60) for applying a voltage potential between the probe (22) and the sample.

5. A probe (22) as claimed in any one of the preceding claims characterised in that the biasing element is provided adjacent the probe tip (4).

6. A probe (22) as claimed in any one of the preceding claims characterised in that the force sensing member (3) has a low quality factor for one or more vibrating modes of the force sensing member (3).

7. A probe (22) as claimed in claim 6 characterised by the force sensing

member (3) including a damping element (22c) adapted to dissipate energy that would otherwise be mechanically stored in the force sensing element through excitation of one or more oscillatory modes.

8. A probe (22) as claimed in claim 7 characterised in that the damping element (22c) comprises a coating of an energy-absorbing material on at least one side of the force sensing element (3).

9. A probe (22) according to claim 8 characterised in that the energy-absorbing material is a polymer film.

10. A probe (22) according to claim 9 characterised in that the polymer film is formed of a copolymer with majority component that is an amorphous rubber and a minority crystalline or glassy component.

11. A probe (22) according to claims 9 or 10 characterised in that the force sensing member (3) is coated with polymer by solution casting.

12. An atomic force microscope (10) for imaging a sample in accordance with an interaction force between the sample and a probe (22), the microscope (10) comprising

driving means (16, 18, 20, 70) arranged to provide relative scanning motion between the probe (22) and the sample surface and capable of bringing the sample and probe (22) into close proximity, sufficient for a detectable interaction to be established between them; and

a probe detection mechanism (28) arranged to measure deflection and / or displacement of the probe (22);

characterised in that, the microscope (10) includes the probe (22) of any one of claims 1 to 11.

13. An atomic force microscope as claimed in claim 12 characterised by further comprising a resonant oscillator mechanically coupled to either the probe (22) or a sample stage for causing relative oscillatory movement between the probe (22) and the sample.

14. An atomic force microscope (10) for imaging a sample in accordance with an interaction force between the sample and a probe (22), the microscope (10) comprising

driving means (16, 18, 20, 70) arranged to provide relative scanning motion between the probe (22) and the sample surface and capable of bringing the sample and probe (22) into close proximity, sufficient for a detectable interaction to be established between them; and

a probe detection mechanism (28) arranged to measure deflection and / or displacement of the probe (22);

characterised in that, the microscope (10) includes force (F_{direct}) generating means (24, 26; 50, 60) arranged such that, in operation, a force (F_{direct}) is applied to either or both of the sample and the probe (22) or between the sample and the probe (22), the force (F_{direct}) being directed so as to urge the probe (22) towards the sample or *vice versa*.

15. A microscope according to claim 14 characterised in that the force (F_{direct}) has a magnitude that is substantially independent of the degree of deflection of the probe (22).

16. A microscope according to claim 15 characterised in that the probe (22) has spring constant k and the probe (22) properties and the applied force (F_{direct}) are selected such that, at least within a predetermined timescale, the applied force (F_{direct}) is greater than the restoring force kx provided by a deflection x of the probe (22) as it scans the surface of

the sample.

17. A microscope according to claim 16 characterised in that the probe (22) has spring constant k that is less than 1 Nm^{-1}.

18. A microscope according to any one of claims 14 to 17 characterised in that the force (F_{direct}) generating means comprises a magnet (26) and a magnetic element (24) incorporated in the probe (22).

19. A microscope according to any one of claims 14 to 17 characterised in that the force (F_{direct}) generating means comprises means (50, 60) for applying an attractive biasing voltage between the probe tip (4) and the sample.

20. A microscope according to any one of claims 14 to 17 characterised in that the force (F_{direct}) generating means comprises a sample environment which encourages the formation of a capillary neck between the probe (22) and the sample, the capillary neck providing said applied force (F_{direct}).

21. A microscope according to claim 20 characterised in that the force (F_{direct}) generating means further comprises a hydrophilic surface on said probe (22).

22. A microscope according to any one of claims 14 to 21 characterised in that the probe (22) has a low quality factor.

23. A microscope according to claim 22 characterised by further comprising means (80) for immersing the probe (22) and sample in a liquid during operation of the microscope.

24. A microscope according to claim 22 characterised in that the force sensing element (3) of the probe (22) includes a damping element

(22c) adapted to dissipate energy that would otherwise be mechanically stored in the force sensing element (3) through excitation of one or more oscillatory modes.

25. A microscope according to claim 24 characterised in that the damping element comprises a coating of a polymeric material (22c) on at least one side of the force sensing element (3).

26. An atomic force microscope as claimed in any one of claims 14 to 25 characterised by further comprising a resonant oscillator mechanically coupled to either the probe (22) or a sample stage for causing relative oscillatory movement between the probe (22) and the sample.

27. A method of collecting image data from a scan area of a sample with nanometric features wherein the method comprises the steps of:-

(a) moving a probe (22) having a force sensing element (3) with a tip (4) having a tip radius of 100nm or less into close proximity with a sample in order to allow an interaction force to be established between probe (22) and sample;

(b) causing a force (F_{direct}) to be established between sample and probe (22) such that the probe (22) is urged to move towards the sample (14) or *vice versa*;

(c) scanning either the probe (22) across the surface of the sample or the sample beneath the probe (22) whilst providing a relative motion between the probe (22) and sample surface such that an arrangement of scan lines covers the scan area;

(d) measuring deflection and / or displacement of the probe (22); and

(e) processing measurements taken at step (d) in order to extract

information relating to the nanometric structure of the sample.

28. A method as claimed in claim 27 characterised by further comprising during step (c) dissipating energy which otherwise would be stored in the force sensing element (3) through excitation of oscillatory modes.

29. A method as claimed in either claims 27 or 28 characterised in that the relative motion between the probe (22) and the sample surface under step (c) is provided by a resonant oscillator.

30. A scanning probe microscope (10) for writing information to a sample by means of an interaction between the sample and an AFM cantilever probe (22), the microscope comprising

driving means (16, 18, 20, 70) arranged to provide relative scanning motion between the probe (22) and the sample surface and capable of bringing the sample and probe (22) into close proximity; and

a probe writing mechanism arranged to vary intermittently, typically on a timescale shorter than one scan line, the strength of the interaction between the probe and the sample and so to change intermittently a property of the sample surface in the locality of the probe;

characterised in that, the microscope (10) includes force (F_{direct}) generating means (24, 26; 50, 60) arranged such that, in operation, a force (F_{direct}) is applied to either or both of the sample and the probe (22) or between the sample and the probe (22), the force (F_{direct}) being directed so as to urge the probe (22) towards the sample or *vice versa*.

31. A method as claimed in claim 30 characterised in that the relative motion between the probe (22) and the sample surface is provided by a resonant oscillator.